

A STUDY OF INTERLAYER GALLERY OF MODIFIED SODIUM
MONTMORILLONITE (MMT) NANOFILLER USING ION EXCHANGE AND
SURFACTANT METHOD

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Thesis submitted in partial
fulfillment of the requirements for the award of the
degree of Bachelor of Chemical Engineering

Faculty of Chemical and Natural Resources Engineering
UNIVERSITI MALAYSIA PAHANG

FEBRUARY 2012

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ABSTRACT

Polymer/layered-silicate hybrid nanocomposites have attracted strong interest in today's materials research, as it is possible to achieve impressive enhancements of material properties compared to the pure polymers. This study aim is to modify the clay surface using ion exchange and surfactant methods before proceeding with fabrication of polymer nanocomposites. The polymer that used is Polylactic acid (PLA) because of its biodegradability characteristic. Organoclay will characterize by using Atomic Adsorption Spectrometry (AAS) and Fourier Transform Infrared Spectroscopy (FTIR). PLA-Nanocomposites were prepared by using solution casting and characterized by X-Ray Diffraction Analysis (XRD), Scanning Electron Microscopy (SEM). AAS analysis showed there is a transition metal ion in the interlayer of sodium montmorillonite (MMT) and it is confirmed with SEM analysis. The FTIR analysis for surfactant organoclay shows the presence of TEOS surfactant in the organoclay properties. The XRD analysis proved that the modification happens by the increasing of clay interlayer basal spacing. The XRD analysis for PLA-Nanocomposites showed that PLA is intercalated the interlayer gallery of organoclay to similar extents and also increased the d-spacing. The structure of polymer nanocomposites that came out of this research is intercalated and exfoliated nanocomposites.

Keywords: surface modification, ion exchange method, surfactant method, PLA-nanocomposites.

KAJIAN TENTANG MODIFIKASI STRUKTUR DALAMAN TANAH LIAT DENGAN MENGGUNAKAN KAEDAH PERTUKARAN ION DAN SURFAKTAN.

ABSTRAK

Pada waktu ini, kajian tentang polimer nano komposit telah menarik minat ramai penyelidik bahan kerana ia memberi banyak kelebihan berbanding komposit biasa. Target kajian ini adalah memodifikasi struktur dalaman tanah liat dengan menggunakan kaedah pertukaran ion dan surfaktan. PLA digunakan sebagai polimer kerana sifatnya yang mudah terurai. Ciri-ciri organoclay akan ditentukan dengan menggunakan Atomic Adsorption Spectrometry (AAS) dan Fourier Transform Infrared Spectroscopy (FTIR). PLA-Nanokomposit dihasilkan dengan menggunakan kaedah solution casting dan X-Ray Diffraction Analysis (XRD), Scanning Electron Microscopy (SEM) digunakan untuk menentukan ciri-ciri nya. AAS analisis menunjukkan kehadiran ion besi didalam struktur dalaman organoclay dan ia disahkan oleh analisis SEM. Analisis FTIR yang dijalankan keatas sampel organoclay surfaktan menunjukkan kehadiran surfaktan TEOS didalam struktur organoclay. Analisis XRD membuktikan bahawa modifikasi menyebabkan jarak antara dua struktur dalaman tanah liat bertambah. Analisis XRD pada sample PLA-Nanokomposit menunjukkan PLA meresap kedalam struktur dalaman tanah liat dan menyebabkan pertambahan jarak antara dua struktur dalaman tanah liat.

Kata kunci: modifikasi permukaan, kaedah pertukaran ion, kaedah surfaktan, PLA-Nanokomposite.

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CHAPTER 1

BACKGROUND OF THE RESEARCH

1.1 Introduction

Polymer/layered-silicate hybrid nanocomposites have attracted strong interest in today's materials research, as it is possible to achieve impressive enhancements of material properties compared to the pure polymers (Strawhecker et. al., 2000). These composites exhibit superior strength, modulus, thermal resistance and heat distortion temperature (Joshi et. al., 2004). The first step before proceed with fabrication of polymer nanocomposites is to modify the surface of nanoclay.

The main reason of surface modification of nanoclay is to ensure that nanoclay is compatible with polymer matrix. According to Qian et. al. (2008), in his experimental work, he looks forward to focusing on how to modify the surface of nanoclay that enhancing the interaction between clay surface and polymer chain and by using a sol-gel modification approach, it combines the surface properties of mesoporous silica and nanoparticles with layered clay, while inheriting the structural properties of the pristine clay

such as further intercalation with organic compounds and polymers. One of the drawbacks of clays is the incompatibility between hydrophilic clay and hydrophobic polymer, which often causes agglomeration of clay mineral in the polymer matrix. Therefore, surface modification of clay minerals is the most important step to achieve polymer nanocomposites. Upon organic treatment, clays become hydrophobic and hence compatible with the polymers (Zheng et. al., 2005)

Clay is the most common layer solid that are used widely. Based on Zheng et. al. (2005), there is many types of layered solids such as graphite, metal phosphates, etc. But the most common used in researches and industry are a clay mineral because of the uniqueness of its structure and properties. There are many types of polymer that can be used and Poly (lactic acid) polymer (PLA) is chosen in this study. Vahik et. al. (2003) state that Poly (lactic acid) polymer (PLA) is one of example of polymer that commonly used for packaging because of its biodegradability and for sure is ecologically friendly. According to McLauchlin et. Al. (2009), PLA is produced by fermenting dextrose derived from maize.

In surface modification of clay, the sodium montmorillonite have a hydrophilic interlayer characteristic while polymer has a hydrophobic interlayer characteristic. So, by using an organic treatment, the clay will be treat into hydrophobic characteristics to make sure it's compatible with polymer. According to Zheng et. al. (2005), it is important to change the inorganic clay to organic clay in the modification of nanocomposites in order to get a perfect compatibility with the polymer. Nowadays, many researchers focus on how to increase the exfoliation of the clay platelets in polymer matrixes by enhancing interaction between clay surface and polymer chains (Qian et. al, 2008). There two types of surface modification that we study here is ion exchange and surfactant method.

In the ion exchange method, the hydrophilic silicate of clay needed to be converted into hydrophobic in order to layer silicates miscible with most polymer matrices. This can

be done by ion exchange modification with cationic surfactants. Clay layer is stacked together with van der Waals to form a gallery. By substituting lower valent metallic ion for Si, Al, or Mg in the layer will generate negative charges that are counteracted by the alkali and alkaline cation within the galleries as according to Liu et. al. 2006. Nawani et. al. (2007) said that the ion exchange method can be done by react the pristine clay with the Transition Metal Ion salt (TMIs) in the presence of solvent as shown in **Table 1.1**.

Table 1.1: TMI Salts and Solvents Used in the Modification Procedure (Pranav et. al, 2007)

Type of clay	A solvent used	TMI salt used	Concentration [M]
Sodium Montmorillonite (Pristine clay)	Water	Copper (II) sulfate	0.3
	Methanol	Copper (II)chloride	0.3
	Ethanol	Copper (II)chloride	0.3
	Dioxane	Copper (II) Nitrate	0.3

Different with surfactant method, Na^+ MMT will be modified with cationic surfactants (eg:, alkylammonium cation) to lower the surface energy and improve wetting characteristic of clay. So, intercalation is possible. This cation also provides a functional group that can react with polymer matrix or initiate polymerization to strengthen the interface between inorganic filler and an organic polymer matrix (Liu et. al., 2006).

For the dual surfactant method, recent studied by Andrew et. al. (2009) highlighted that mineral needs to modify with a thermally stable surfactant (eg: - aminopropyl-POSS

(AP-POSS)). For example, in dual surfactant method, poly (butylenes terephthalate) (PBT) and ditallowdimethylammonium chloride is sequentially modified with sodium montmorillonite (Na^+MMT) by the ion exchange reaction that will give a dual-surfactant organoclay (OC) (Andrew et, al., 2010).

1.2 Problem Statement

The main problem in polymer-organoclay nanocomposites is incompatibility between polymer and clay. This is due to clay characteristic is hydrophilic while the polymer is hydrophobic. In order to solve this problem, clay characteristic has to be modified into hydrophobic before reacting with a polymer to form nanocomposite. This surface modification will increase the d-spacing of clay interlayer and form a perfect compatibility with the polymer.

1.3 Objective

The first objective of this study is to modify the surface of sodium montmorillonite (Na^+MMT) by using the ion exchange method and surfactant method. The second objective is to fabricate PLA-Nanocomposites by using organoclay from ion exchange and surfactant method with PLA polymer. The third objective is to study the interlayer properties, structure and morphology of organoclay and PLA-Nanocomposites by using these two methods.

1.4 Scope of Study

In order to achieve the objectives, the scope of study is narrowed down to the following:-

- 1) a) To modify the surface of Na^+MMT with Copper (II) Sulfate or Copper (II) Chloride TMI salt in the presence of methanol or ethanol solvent.
b) To modify the surface of mineral clay, Na^+MMT by reacting it with Tetraethyl orthosilicate (TEOS) / alkylammonium to form an organoclay.
- 2) To study which type modification of nanoclay that can provide a good interlayer d-spacing for polymer attachment and also more compatible with polymer.
- 3) To fabricate pla-nanocomposite using solution casting method.
- 4) To compare the structure of pla-nanocomposite from ion exchange method organoclay and surfactant method organoclay by using Scanning Electron Microscopy (SEM) and X-ray diffraction (X-RD).

1.5 Rational and Significant

The point of this experiment is to find out the most compatible surface modification method that can provide a suitable condition for the polymer chain to fill the interlayer space of organoclay because the problem that the issues here is PLA and Na^+MMT are not compatible. Using the following methods, ion exchange and surfactant modification show an improvement on interlayer structure or properties of Na^+MMT . Ion exchange shows improvement by increasing the d-spacing of the interlayer. While surfactant method makes an improvement by providing functional group to interact polar region of PLA chain. The structure of PLA-Nanocomposites shows that exfoliation is occurring.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction to Nanoclay and Polylactic acid (PLA).

2.1.1 Nanoclay/ Pristine Clay/ Sodium Montmorillonite.

One general way to prepare a polymer nanocomposite is by the intercalation chemistry of layered inorganic solids in which polymer is inserted into the interlayer space. There are many types of layer inorganic solid such as graphite, clay minerals, transition metal dichalcogenides, metal phosphates and phosphonates, etc. The common type of layer inorganic solid that used today in industries is clay mineral. The reason of clay mineral widely used in industries is because of its unique structure and properties. Clay minerals have a higher intercalation chemistry ability as compared to other layered inorganic solid when applied with polymer to form polymer nanocomposite. In details, clay mineral's unique layered structure and high intercalation capabilities that allow them to chemically modified to compactable with polymer and became clay-based polymer nanocomposites.

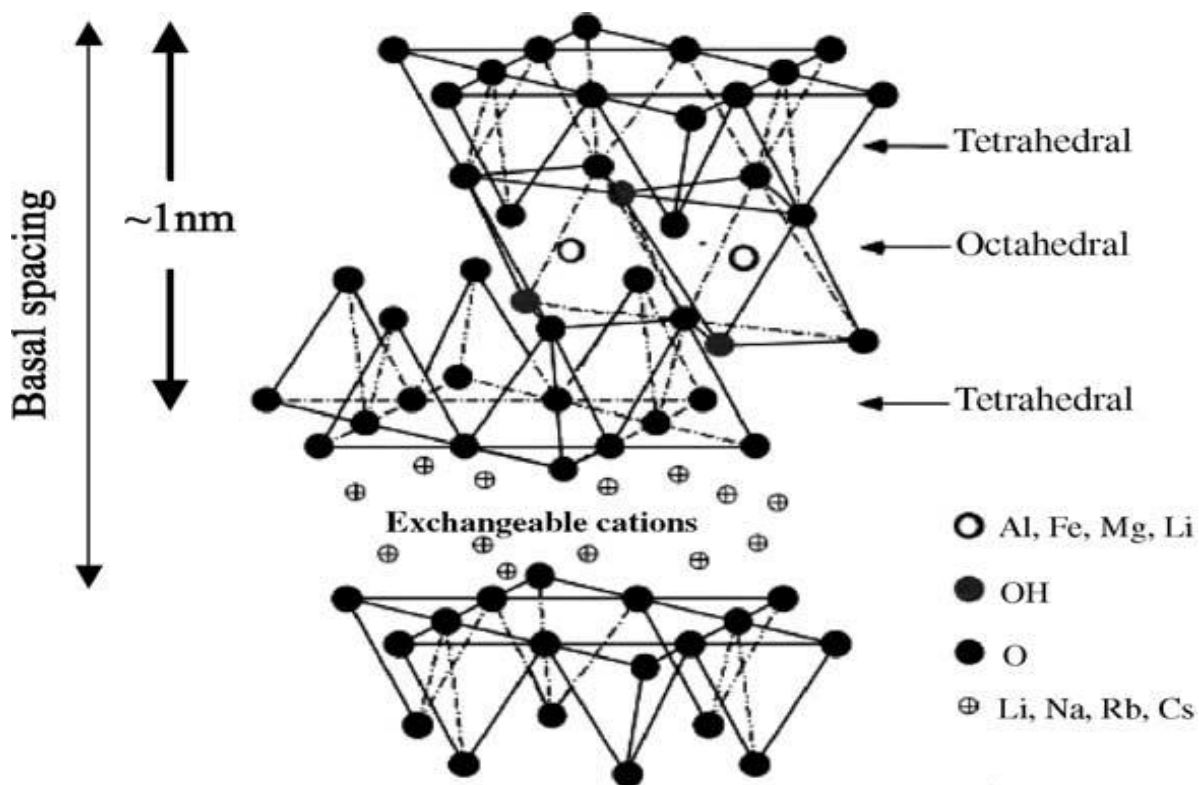


Figure 2.1: Structure of layered silicates (Giannelis, 1999)

As shown in the **Figure 2.1**, clay mineral structure is consisting of interlayer or gallery and this interlayer is caused by the van der Waals force between the layer gaps. This layer gap is made up of two tetrahedrally coordinated silicon atoms fused to an edge-shared octahedral sheet of either aluminum or magnesium hydroxide. There is an isomorphic substitution within the layer for (e.g., Li, Na, Rb and Cs) and also a relatively layer charge. Clay mineral has a relatively low layer charge which is between ($x = 0.2-0.6$). So, the force between the adjacent layer also relatively low and finally the interlayer is cations exchangeable and the intercalation of inorganic and organic cations and molecules into the interlayer space are accomplished. Other advantages of using clay as a layered inorganic solid is it can form a high strength and stiffness polymer nanocomposites and clay is abundance and easy to get and also cheaper compare to other layered organic solids.

Clay mineral can be divided into two types which is natural clay and synthesis clay. The commonly used clay mineral is natural clay. The two types of natural clay that widely used is montmorillonite and hectorite type.

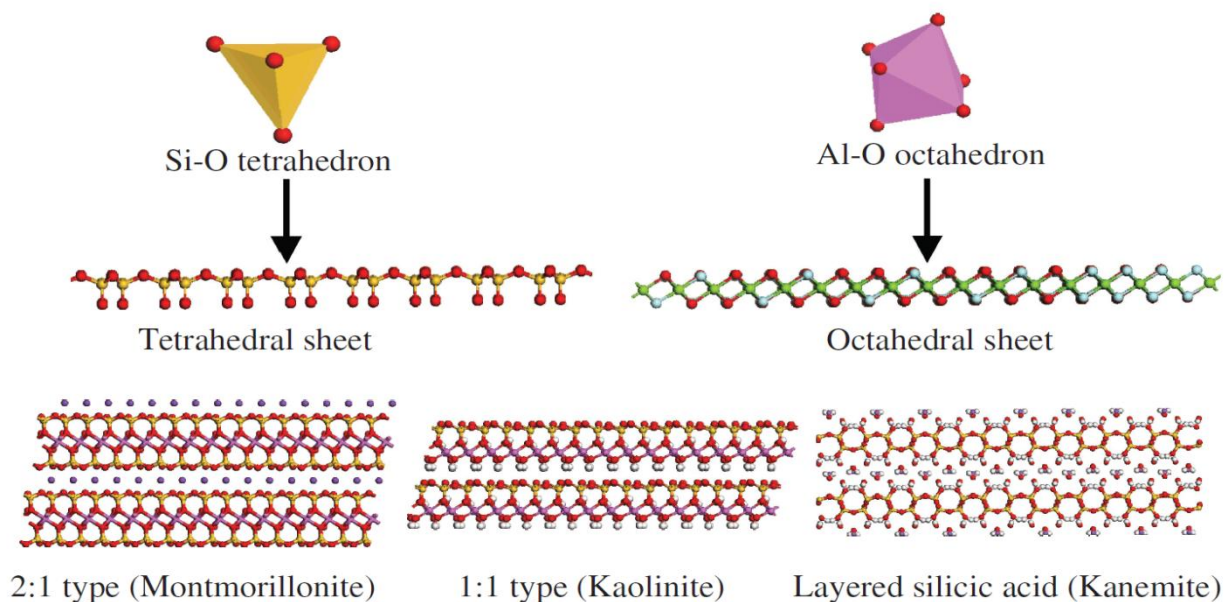


Figure 2.2: Structure of clay minerals represented by montmorillonite, kaolinite, and kanemite.

This two type of clay is falling under 2:1 type of clay. There is also 1:1 type and layered silicic acid type of clay (**Figure 2.2 and Table 2.1**).

The 2:1 type has an aluminium octahedron sheet that sandwiched between two adjacent silicon tetrahedron sheets. Stacking of the layers leads to a van der Waals gap between this layers. Zeng et. al. (2005) mentioned that the isomorphic substitution of Al with Mg, Fe, Li in the octahedron sheets and/or Si with Al in tetrahedron sheets gives each three sheet layer an overall negative charge, which is counterbalanced by exchangeable metal cation residing the interlayer space, such as Na, Ca, Mg, FE, and Li.

The 1:1 type has a layer that made up of one aluminum octahedron sheet and one silicon tetrahedron sheet. The difference of this type of clay with 2:1 type is there was no isomorphic substitution in either octahedron or a tetrahedron. Water molecules the only ion that occupy the space between the layers. This layer is stacked together by hydrogen bonding between hydroxyl groups in the octahedral sheets and oxygen in the tetrahedral sheets of the adjacency.

The Layer Silicic Acids consists of silicon tetrahedron sheets with different layer thickness. Their basic structure composed of layered silicate networks and interlayer hydrated alkali metal cations. Layered silicic acids also are suitable to use for polymer nanocomposites because it has similar intercalation chemistry with 2:1 type clay. It also has high purity and structural properties that are complementary to smectie clays. They are built up from combinations of tetrahedral and octahedral sheets whose basic units are usually Si-O tetrahedron and Al-O octahedron, respectively. (Zeng et. al, 2005)

Table 2.1: Clay mineral used for polymer nanocomposites (Zeng et. al, 2005)

Type of clay	Origin	Substitution	Layer charge
2:1 type:			
MMT	Nature	Octahedral	Negative
Hectorite	Nature	Octahedral	Negative
Saponite	Nature	Tetrahedral	Negative
1:1 type:			
Kaoinite	Nature	NO	Neutral
Halloysite	Nature	NO	Neutral
Layered silicic acid:			
Kanemite	Natural/ Synthetic	Tetrahedral	Negative

2.1.2 Polylactic acid (PLA)

Today, plastic is produced by using fossil fuel and when this plastic is discarding into the environment, it will end up with waste that cannot be degraded spontaneously. In order to dispose this waste, incineration process need be done. This process actually will produce carbon dioxide that can harm the environment in which cause a global warming. Furthermore, incineration also releases toxic gases that also harm to human life (Donald, 2001).

According to the Municipal Solid Waste report issued by the U.S. Environmental Protection Agency (EPA) for the year 2008, 76.76 million tons of wastes were generated from packaging materials. This included 13.01 million tons of plastics with 4.89 million tons of plastic packaging in the form of films. Only 13.2% of this plastic packaging waste was recycled, while only 9.8% of plastic films were recovered; the rest was added to landfills (Samer et. al., 2011)

To solve this problem, researchers have found out that Polylactic acid (PLA) is suitable material to form a plastic because of its renewable resource and most important criteria is readily biodegradable. According to Ray et. al. (2003) this green polymer material did not need toxic component in manufacture and also can degrade in the natural composting process.

Polylactic acid (PLA) is made up of α -hydroxyl acids, which include polyglycolic acid or polymandelic acid. PLA falls under aliphatic polyesters family. PLA characteristic is thermoplastic, high strength, and high modulus. Major use of a PLA is for industrial packaging field or biocompatible or bio-absorbable medical device market. High molecular weight PLA is resistant to support bacterial and fungal growth in food packaging industries. PLA degradable is by simple hydrolysis of the ester bond and do not require an enzyme to speed up the rate of hydrolysis.

